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National Environmental Testing

Dayton Division

Standard Operating Procedure

Analyte or Suite: Aqueous Semi-volatile Sample Preparation

Methodology: Continuous Liquid-Liquid Extraction

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1. SCOPE AND APPLICATION

1.1. This method applies to the isolation and concentration of a number of organic compounds that are partitioned into an organic solvent from an aqueous medium and are amenable to gas chromatography.

1.2. This method is applicable for all aqueous sample types such as groundwater, effluents and surface water.

1.3. This method may be extended to include any compound that is insoluble (or only slightly soluble under the extraction conditions) in a aqueous medium and is sufficiently stable and volatile to elute from a gas chromatograph.

2. SUMMARY OF METHOD

A measured volume of sample, usually one liter, is placed in a continuous liquid-liquid extractor and extracted with Methylene chloride. The extract is dried, concentrated, and packaged for analysis.

3. SAFETY

Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards, and all associated protective wear and spill clean-up procedures PRIOR TO THE USE of any chemical. In all cases, both the applicable MSDS and supervisor or Safety Officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxics, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and labcoats must be worn, and solvents will be handled in ventilated hoods, in addition to other measures prescribed by the Division. It should be noted that samples must be handled with as much (or more) care as any of the materials used in this method due to the unknown nature of their composition. Also, the equipment utilized by this method contains areas of high temperature and voltage.

4.0 REAGENTS AND MATERIALS

The following apparatus and materials, or their equivalent, are required. Reagents and materials are considered equivalent if with their use, the analytical and QA/QC requirements in this SOP can be met.

4.1 Materials

4.1.1. Sample container - glass container equipped with a Teflon lined cap which will hold a minimum of one liter. Sample containers must be purchased with a manufacturers certification of cleanliness for the intended use (e.g. Eagle-Picher 112-01A or I-Chem 349-1000).

4.1.2. Continuous liquid-liquid extractor: Equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication.

4.1.3. Long stem glass funnel. Approximately 2" in diameter.

4.1.4. Kuderna-Danish (K-D) apparatus:

4.1.4.1. Concentrator tube: 10 mL, graduated (Kontes K-570050-1025 or equivalent). Ground-glass stopper is used to prevent evaporation of extracts.

4.1.4.2. Evaporation flask: 250 or 500 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with clips.

4.1.4.3. Snyder column: Three-ball macro (Kontes K-503000-0121 or equivalent).

4.1.4.4. Snyder column: Two-ball micro (Kontes K-569001-0219 or equivalent).

4.1.4.5. Boiling beads.

4.1.5. Water bath: Heated, capable of temperature control (±5°C). The bath should be used in a hood.

4.1.6. Vials: Amber Glass, 1 1/2 mL capacity with Teflon-lined crimp caps.

4.1.7. pH indicator paper; wide range.

4.1.8. Balance - Analytical, capable of accurately weighing to 0.001 g.

4.1.9. Rapid-Vap Nitrogen Blow-Down automatic concentrator.

4.1.10. Miscellaneous: funnels, beakers, disposable pipets, clips, Teflon joint sleeves, microsyringes.

4.2 Chemical Standards

Caution: The following standards may contain one or more known or suspected carcinogens. Read all precautionary information supplied with the standards. Gloves and safety glasses should be worn to avoid contact with eyes, and skin. Any use of these standards in a manner that may cause the release of vapors into the laboratory atmosphere should be conducted within a fume hood. Hoods are classified as designated areas when working with carcinogens.

4.2.1. Standard storage. All standards, spikes and surrogates should be stored at -20°C to -10°C in a separate refrigerator from samples, VOA standards, and VOA samples. Storing extracts at these temperatures will cause some of the components to precipitate. ALWAYS sonicate standard solutions prior to removing aliquots to ensure all components are dissolved. Sonicate for 1 to 3 minutes depending upon the volume of the standard. Visually inspect the standard for precipitate and continue to sonicate if any is present. Standards should be stored in the smallest screw top vial possible to reduce the headspace above the solution. As a standard is used, transfer it to a smaller vial.

4.2.2. Manufacturers of standards sometimes will change the concentration and composition of standards. ALWAYS read the literature provided with the standard so that the concentration and composition of the standard is known. Whoever creates a standard is responsible for informing other analysts of changes in the standard concentration or composition. Prior to creating each standard, the analyst should calculate the final concentration of the standard. Do not use the volumes recorded in the standard logbook for the previous standard assuming the volumes required are the same.

4.2.3. Each standard used must be traceable to the manufacturer lot number. Include all pertinent information for each solution in your Standards Log Book including the date prepared or received, lot number, volume used for dilution, final volume, solvent, catalog number, final concentration, and your initials.

4.2.4. Label each solution vial with the date, standard name, concentration, Standards Log Book Reference Number and your initials.

4.2.5. BNA surrogate solution, special order from Supelco, quote 1441. Concentration of Acid surrogates is 5000 ug/mL. The

concentration of Base Neutral surrogates is 2500 ug/mL.

4.2.6. Standards for BNA Matrix Spike and LCS Solutions

4.2.6.1. Restek SV Cal Mix 1. Catalog #11860. 2000 ug/mL.

4.2.6.2. Restek SV Cal Mix 2. Catalog #11900. 2000 ug/mL.

4.2.6.3. Restek SV Cal Mix 3. Catalog #11940. 2000 ug/mL.

4.2.6.4. Restek SV Cal Mix 4. Catalog #11980. 2000 ug/mL.

4.2.6.5. Restek SV Cal Mix 5. Catalog #12020. 2000 ug/mL.

4.2.6.6. Restek SV Cal Mix 7. Catalog #12080. 2000 ug/mL.

4.2.6.7. Restek 3,3'-Dichlorobenzidine Catalog #11230 at 2000 ug/mL.

4.3. Working Spike Solutions

4.3.1 Working BNA surrogate spike solutions. Add approximately 150 mL of purge and trap Methanol to a 200 mL volumetric flask. Add 8 mL of the Supelco Surrogate mix to the volumetric flask and dilute to volume with Methanol. Invert slowly three times to mix. The solution then contains each of the Base-Neutral compounds at 100 mg/L and the Acidic compounds at 200 mg/L.

4.3.2. Working BNA Spike Solutions

4.3.2.1 The Spiking Solution for LCSs and MS/MSDs is prepared as described below in a 10 mL volumetric flask. Add approximately 2 mL of Methylene chloride to the flask. Add the amounts specified below to the solvent in the 10.0 mL volumetric flasks. Use a 1.0 mL syringe with a fixed needle to add these volumes. Dilute to the mark with Methylene chloride. Invert slowly three times to mix.

TABLE 1.

LCS and MS/MSD Spiking Solution	Catalog	Amount
Restek Cal Mix 1	11860	1.0 mL
Restek Cal Mix 2	11900	1.0 mL
Restek Cal Mix 3	11940	1.0 mL
Restek Cal Mix 4	11980	1.0 mL
Restek Cal Mix 5	12020	1.0 mL
Restek Cal Mix 7	12080	1.0 mL
Restek 3,3'-Dichlorobenzidine	11230	1.0 mL

4.3.2.2. The final concentration of these solutions is 200 mg/L. Spike 0.5 mL of each solution per BNA Matrix Spike (MS), Matrix Spike Duplicate (MSD), and Laboratory Control Sample (LCS).

4.3.2.3. Transfer the solution above to 1 1/2 mL amber vials. As the standards are used transfer them to smaller vials to reduce the amount of headspace in the vial. Discard the standard after six months.

4.4 Reagents

4.4.1. Reagent water: Reagent water is defined as water in which an interference is not observed at the method detection limit of the compounds of interest.

4.4.2. Sodium sulfate: (ACS) Granular anhydrous (purified by washing with Methylene chloride followed by heating at 400°C for 4 hr in a shallow tray).

4.4.3. Methylene chloride - Pesticide quality or equivalent.

Caution: Methylene chloride is a suspect carcinogen. Gloves and safety glasses should be worn to avoid contact with eyes and skin. Avoid inhalation by working with Methylene chloride in a fume hood. Any use of Methylene chloride that may cause the release of vapors into the laboratory atmosphere should be conducted within a fume hood. Hoods are classified as designated areas when working with carcinogens.

4.4.4. Acetone - Pesticide quality or equivalent.

Caution: Acetone is a dangerous fire hazard. Acetone may explode when exposed to heat. Keep away from heat, sparks, and flames. Acetone is a skin, and eye irritant. Gloves and safety glasses should be worn to avoid contact with eyes and skin. Avoid inhalation by working with this solvent in a fume hood. Acetone is a narcotic when ingested.

4.4.5. Methanol- Pesticide quality or equivalent.

Caution: Methanol (Methyl alcohol) is toxic by ingestion, inhalation, and absorption. Gloves and safety glasses should be worn to avoid contact with eyes and skin. Avoid inhalation by working with this solvent in a fume hood.

4.4.6. Sulfuric Acid Solution (1:1 about 18N) - Reagent Grade.

Slowly add 500 mL of H2S04 (sp. gr. 1.84) to 500 mL of reagent water. If method blanks indicate contamination, this solution should be purified by extracting with Methylene chloride. (ACS grade e.g. Baker Analyzed)

Caution: Sulfuric Acid is poisonous-may be fatal if swallowed. Can cause severe burns. Avoid contact with eyes, skin, clothing. Wash after working with Sulfuric acid. When diluting with water, always add the acid to the water so that the concentration of acid increases slowly. If a small amount of water is added to a large quantity of acid, there may be a violent reaction which may generate sufficient heat to ignite combustible materials (or solvents). Avoid breathing vapors. Keep in tightly closed container. Work with Sulfuric acid in a fume hood. As always, gloves, safety glasses, and lab coat are required to be worn.

4.4.7. Sodium hydroxide solution (6N) - Reagent Grade.

Dissolve 60 g dry NaOH pellets into 250 ml of reagent water. If method blanks indicate contamination, this solution should be purified by extracting with Methylene chloride. (ACS grade e.g. Baker Analyzed)

Caution: Sodium hydroxide is poisonous-may be fatal if swallowed. Can cause severe burns. Avoid contact with eyes, skin, clothing. Wash after working with Sodium hydroxide. If a small amount of water is added to a large quantity of Sodium hydroxide, there may be a violent reaction which may generate hydrogen gas and ignite. May also generate sufficient heat to ignite combustible materials (or solvents). Avoid breathing vapors. Keep in tightly closed container. Work with Sodium hydroxide in a fume hood. As always, gloves, safety glasses, and lab coat are required to be worn.

5. INTERFERENCES

5.1 Preliminary set-up. Ensure all equipment and glassware in the following sections are available, in functional order and clean.

5.2. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by extracting Method Blanks.

5.3. Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. This should be followed by washing with Alconox in hot water, and rinses with tap water and reagent water followed by Acetone then Methylene chloride. It should then be drained, and dried. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store it inverted or capped with aluminum foil. At no point in the cleaning of the glassware, should it be rinsed with a solvent (such as Acetone) from any type of plastic rinse bottle. Any solvent rinsing should be done simply by using a Pasteur pipet with the solvent in a glass beaker or erlenmeyer. Also, the glassware should not be washed in any type of plastic tray.

5.4. The use of high purity reagents and solvents helps to minimize interference problems.

5.5. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industry, municipality or hazardous waste site.

6. PROCEDURE

6.1. A measured aliquot of sample (approximately 1 L) is placed into a continuous extractor, and surrogate spike compounds are added to it. The sample is then extracted with Methylene chloride for 18 hours to 24 hours. This extract is then dried, concentrated to a final volume of 1.0 mL, and packaged for analysis.

6.2. The samples must be refrigerated at 4°C from the time of receipt until extraction.

6.3. All extractions must be initiated within seven days of sample collection. If this holding time has been exceeded, notify your Supervisor before proceeding.

6.4. Ready all glassware required for the number of samples to be extracted.

6.5. If not already performed, rinse down all glassware and Sodium sulfate with a small amount of Methylene chloride to remove any possible interferences.

6.6. Log all sample, spikes and blanks into the BNA L/L Aqueous Prep Record book.

6.7. Extraction

6.7.1. Record the date, sample number, sample volume and any observations about the sample in the bound extractions notebook in pen.

6.7.2. For Base/Neutral ONLY extractions proceed with glassware set-up (6.7.3) followed by the extraction (6.7.4.). For Acid ONLY extractions, proceed with glassware set-up (6.7.3) followed by the extraction (6.7.5.). For a full BNA extraction, proceed with sections 6.7.3 through 6.7.5.

6.7.3. Continuous L/L Glassware Preparation

6.7.3.1. Add 550 mL of Methylene chloride to the extractor flask using a graduated cylinder in a hood.

6.7.3.2. Add a magnetic stir star to the extractor flask.

6.7.3.3. Connect the extractor side arm to the extractor flask making sure that the connection is firm.

6.7.3.4. Place the 250 mL collection flask in the heating mantle and add several suitable boiling chips or beads. Connect this flask to the side arm making sure that the connection is firm.

6.7.4. Base Neutral Extraction

6.7.4.1. Mark the water meniscus in the side of the sample bottle for later determination of sample volume.

6.7.4.2. Check the pH of the sample with wide-range pH paper and then adjust the pH to >11 with 6N NaOH.

6.7.4.3. Slowly pour the sample into the extractor with the aid of a long stemmed glass funnel taking care not to allow the sample to run over into the boiling flask via the top sidearm or reach the lower solvent transfer arm.

6.7.4.4. Rinse out the sample bottle with approximately 60 mL of Methylene chloride and add this to the extractor.

6.7.4.5. Add enough reagent water to fill the large round bottom flask so that the solvent just begins to transfer to the small round bottom flask.

6.7.4.6. Add 1.0 mL of the BNA surrogate solution to each sample, blank or spike.

6.7.4.7. Determine the sample volume utilized for extraction by adding DI water to the meniscus mark on the sample bottle. Pour

the water into a 1 L graduated cylinder and record the volume.

6.7.4.8. After extracting for 1 hour, turn the extractors off and let the system cool. Check the pH of the sample with wide range pH paper. The pH should be >11. Adjust if necessary with 6N NaOH. Resume the extraction.

6.7.4.9. Allow the extraction to continue for a total of 18-24 hours.

6.7.4.10. If Base Neutral analysis alone is required, cool and remove the boiling flask. The extract may be stored in a capped boiling flask at 4°C until the next step is performed (6.9).

6.7.4.11. If Acid analysis is also required, continue at Section 6.7.5.3.

6.7.5. Acid Extraction

6.7.5.1. If an Acid only extraction is to be performed follow the Base Neutral extraction procedure 6.7.4. but adjust the pH of the initial sample to <2 by using 1:1 Sulfuric Acid.

6.7.5.2. If the Acid extraction is a continuation of the BNA prep, then continue with this section.

6.7.5.3. Following the BN extraction from the previous day, turn off the heating mantles and allow the system to cool.

6.7.5.4. Place a clean 250 ml collection flask in the heating mantle. Add several suitable boiling chips or beads and approximately 100 ml of Methylene Chloride. Connect this flask to the sidearm, making sure that the connection is tight.

6.7.5.5. Remove the condenser from the L/L side arm and add 40 mL of 1:1 Sulfuric acid to the column of aqueous sample. Be careful that no acid goes down the side arm into the boiling flask as this will damage the sample and a repreparation will be called for. Reposition the condenser and continue the extraction.

6.7.5.6. After extracting for 1 hour, turn the extractors off and let the system cool. Check the pH of the sample with wide range pH paper. The pH should be <2. Adjust the pH with 1:1 sulfuric acid if necessary and resume the extraction.

6.7.5.7. After a total of 18-24 hours, allow to cool and then remove the boiling flask. The extract may be stored in a capped boiling flask at 4°C until the next step is performed (6.9).

6.8. MS/MSD, LCS, and Method Blank Extraction.

6.8.1. To assess the effectiveness of the procedure on various matrices, "spiking" of the matrix is performed. A matrix is spiked by adding a known amount of analytes to the matrix prior to the extraction. The percent recovery of the analytes is calculated from the analytical results. Duplicate aliquots of the matrix are spiked so that the precision or reproducibility of the procedure can be assessed. Therefore, three aliquots of one sample are measured for extraction. Two of the aliquots are spiked with a solution containing analytes of a known concentration. These samples are referred to as the Matrix Spike (MS) and the Matrix Spike Duplicate (MSD). The aliquot which is not spiked is referred to as the Matrix Unspiked (MU).

6.8.2. A Laboratory Control Standard (LCS) is extracted to demonstrate ability to perform the extraction and analysis in the absence of a matrix effect. To create an LCS, deionized water is spiked with matrix spike solution and extracted.

Should there not be sufficient sample volume for any sample in the extraction batch to perform an MS/MSD, an LCS/LCS duplicate may be performed in its place. Simply spike an extra aliquot of DI water in the prep batch being performed.

6.8.3. A Method Blank is extracted to demonstrate whether or not any contamination of the samples is introduced during the extraction and analysis process. To create a Method Blank surrogates are added to deionized water and the deionized water is extracted.

6.8.4. The MS/MSD set, LCS, and Method Blank must be extracted, and concentrated in the same manner as the Matrix Unspiked sample. Over a period of time a variety of MS/MSD, LCS and blank extracts will be put through the various extraction, and concentration steps as dictated by the Matrix Unspiked samples. At a minimum, an LCS and Method Blank extraction are required to be performed per each day, per each extraction technique, per each set of 20 samples or less, whichever is more frequent. An MS/MSD is performed 1 per extraction batch of up to 20 samples and does not need to be performed daily.

6.8.5. MS/MSD. Select a sample for spiking. Measure two 1.0 liter volumes of sample as in Section 6.7.4.1. Spike with the matrix spike solution immediately after the surrogates are added in Section 6.7.4.6. The BNA batches must be spiked with 0.5 mL of the BNA spiking solution. Acid or Base Neutral only batches must be spiked with 0.5 mL of the BNA spiking solution even though some of the analytes in the spike will not be evaluated.

6.8.6. It will be necessary to collect samples in triplicate to perform an MS/MSD on them.

6.8.7. LCS. To create an LCS, spike the deionized water in a filled liquid/liquid extractor with 0.5 mL of the Matrix Spike Solution listed in Section 4.3.2.1. Add 1 mL of surrogate spiking solution listed in Section 4.3.1.

6.8.8. Method Blank. Fill a liquid/liquid extractor with deionized water and add 1.0 mL of the surrogate spiking solution listed in section 4.3.1.

6.9. Drying the Extract

6.9.1. Place a long stemmed glass funnel over a 250 mL assembled KD apparatus or Rapid Vap glassware. Place a small tuft of glass wool into the funnel. Fill the funnel with Sodium sulfate to within approximately 2 cm of the top.

6.9.2. Pour the extract through the long stemmed funnel containing Sodium sulfate. Collect the dried Methylene chloride in the concentration glassware.

6.9.3. Rinse the round bottom flask twice with approximately 25 mL of Methylene chloride. Pour the rinsate through the Sodium sulfate, combining the rinsate and extract in the KD flask.

6.10 Sample Concentration

6.10.1. KD Concentration

6.10.1.1. Place one boiling bead in the K-D ampule. Attach the ampule to the K-D flask. Transfer the extract into it following Section 6.9.

6.10.1.2. Prewet the Snyder column by adding about 1 mL of Methylene chloride to the top of the column.

6.10.1.3. Place the K-D apparatus on a hot water bath (60-65°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with water.

6.10.1.4. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15-20 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent.

6.10.1.5. When the apparent volume of liquid reaches 1 to 2 mL, remove the K-D apparatus from the water bath and allow it to drain and cool. The sample must not be allowed to go dry. For all practical purposes the extract is destroyed if this occurs. Therefore, exceptional care must be taken to monitor the concentration step, particularly as the sample volume becomes less than about 10 mL.

6.10.1.6. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL of Methylene chloride. A disposable Pasteur pipet may be used.

6.10.2. Nitrogen Blowdown

6.10.2.1. Use the nitrogen blow-down apparatus to further concentrate the extract. Place the ampule in the apparatus. Adjust the height of the needle to be about one inch above the surface of the sample extract. Set the pressure on the regulator to 5 PSI. Adjust the flow through the apparatus so that the stream of nitrogen penetrates the surface of the extract about a quarter of an inch but without causing splashing of the extract. Record the actual volume of the extract.

6.10.2.2. Once the level of the extract is between 0.7 to 0.9 mL, dilute the sample to 1.0 mL with Methylene chloride using a Pasteur pipet. The "final volume" of the extract is 1.0 mL. Proceed to 6.11.

6.10.2.3. It is not unusual for samples to become dark and even to become viscous as concentrated. For these samples, the analyst should stop the concentration at a final volume greater than 1 mL. This prevents highly contaminated extracts form damaging sensitive GC or GC-MS systems. To make calculations simple, stop at a increments of 1 mL, such as 2.0 mL, 5.0 mL, 10.0 mL and so on. When the extract volume is greater than one, the analyst should pipet 1.0 mL of the extract to a 1-1/2 mL vial. The remaining extract should be stored in a vial. Proceed to step 6.11.

6.10.3. Rapid Vap Concentration

6.10.3.1. Quantitatively transfer the BNA extract to the Rapidvap concentrator tube by pouring the extract through the Sodium sulfate described in section 6.9.

6.10.3.2. Standard settings for Methylene chloride with an extract volume of approximately 200 mL are as follows:

Vortex Speed: 60%
Temperature: 56°C

6.10.3.3. Depending on the volume of sample the vortex speed must be decreased as sample may spill out of the tube. Cover each tube with aluminum foil and tape it in place. Punch a hole in the very center of the aluminum foil to make room for the nitrogen jets.

6.10.3.4. The nitrogen pressure to the Rapidvap should not exceed 10 PSI and should be adjusted at the Nitrogen tank.

6.10.3.5. Place all of the tubes in the Rapidvap and close.

6.10.3.6. On the console, indicate the length of the run as well as indicating the positions which have samples by turning on the nitrogen to those positions only.

6.10.3.7. The Rapidvap will alarm the analyst when the programmed run time has elapsed. But, care should be taken to check the level of the Methylene chloride in the tubes periodically so that the sample will not go dry.

6.10.3.8. When the sample is concentrated to 1 mL, remove the sample from the unit and using the sample concentrate rinse down the inner portion of the concentrator tube and then transfer the sample to a 1 1/2 mL vial. Proceed to step 6.11.

6.11 Packaging the Extract

6.11.1. Using a pipet, transfer the sample extract to a 1-1/2 mL autosampler vial and seal it.

6.11.2. The vials must be labelled with the following information: If the extract is a client sample, include the NET sample number. If it is a matrix spike, add MS or MSD to the sample number (ie 12345MS). Label method blanks (BLK) and Laboratory Control Standards (LCS) as such.

6.11.3. Store the extract at 4°C in a refrigerator or cooler designated for extracts. Do not store the extracts with samples or standards.

7. QUALITY ASSURANCE

7.1. Method blanks are required to be extracted each day samples are extracted. A Method Blank is required per extraction technique, per each set a 20 samples or less, per day. The method blank consists of 1 liter of deionized water extracted in the same manner as the samples. The Blank should contain the same surrogates as the samples.

7.2. One set of Matrix Spike and Matrix Spike Duplicate (MS/MSD) is required per set of 20 samples, per extraction technique. The Matrix Spikes may be replaced with LCS/LCS dups if sample volume does not permit the preparation of a sample MS/MSD.

7.3. One Laboratory Control Sample (LCS) is required to be extracted per set of 20 samples or less, per day, per extraction technique.

8. REFERENCES

8.1. U.S EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act: Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

8.2. Test Methods for Evaluating solid Waste, EPA SW-846, September, 1994, Method 3520B.

8.3 USEPA Contract Laboratory Program Statement of Work for Organics Analysis, 2/88.